High Pressure Inductive Measurements using Anvil Cells

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1. Introduction

Many properties of correlated electron systems can be varied by changing the distance between the constituent atoms. In the tight binding model, the hopping integral increases when this interatomic distance is reduced. This could, for instance, lead to the formation of electronic bands, giving rise to a new, different state. To manipulate the lattice spacing, pressure is a powerful tool; and devices have been developed to exert pressure in a control manner.

Loosely speaking, there are two goals for high pressure research. The first is to discover interesting states by mapping the high pressure phase diagram. This is related to the concept of 'tuning'. By employing a set of tuning parameters such as magnetic field, electric field, doping and pressure, the intricate electronic properties of the system can be 'fine tuned', enabling the identification of exotic phases using the same sample. To this end, it is useful to mention that a tuning parameter that does not break time reversal symmetry is particularly attractive – and pressure is one such parameter – because by careful chemical synthesis, it is possible to engineer materials with the 'right' starting lattice constants to mimic the state of interest. Magnetic field, which breaks time reversal symmetry, does not offer the same luxury – for example, it is a nontrivial exercise to bring fractional quantum Hall state back to zero field.

The second goal of this research is to conduct careful study of the high pressure phases discovered. To fulfil this goal, one needs as many tools as possible in the high pressure toolbox. In addition, it is also appealing to be able to reach pressures that are as high as possible. Therefore, pressure cells utilising diamond or Moissanite anvils are the most common. In this article, I will describe some *inductive* experiments we have performed [1-9] that have allowed us to extract microscopic information of the high pressure phase.

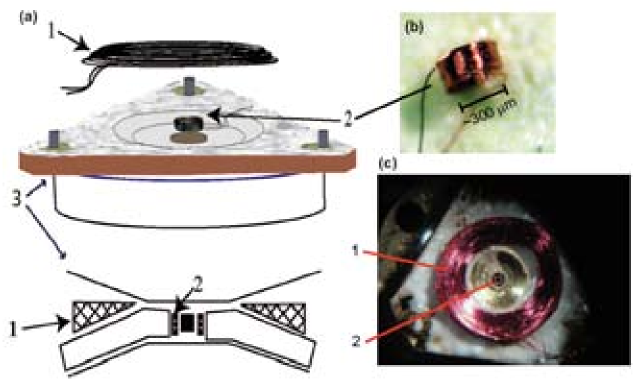

Figure 1. (a) A schematic diagram and (c) a photograph showing the arrangement of the primary driving coil (1), secondary pick-up coil (2) and the anvils (3). (b) A typical pick-up coil with a diameter of 300 μm.

2. The preparation of the pressure cells

To perform inductive measurements, it is highly desirable to have a coil which is only slightly larger than the sample. In other words, the filling factor η, defined as the ratio of the sample volume to the volume of the coil, should be as close to unity as possible. This poses a challenge to high pressure works using anvil cells. To understand this challenge, we have to understand the construction of the sample space for this type of pressure cell. First, a thin (metallic) disk is pressed between the anvils so that it deforms plastically and more or less takes the shape of the anvil. After that, a small hole is drilled at the centre of the deformed region. This 'gasket' hole, bounded at the top and bottom by the anvils, is the sample space. Therefore, if a coil is placed around the anvil, one achieves a typical filling factor of ~10^{-6}. To complicate the situation, the gasket material contributes background signals, making it difficult to interpret the data obtained.

To overcome these issues, Alireza and Julian took a bold approach – they developed a technique to place a tiny coil *inside* the gasket hole [10]. Briefly, a 10-turn coil with a diameter of about 300 μm was wound using small insulated copper wire. Two channels were carved on the gasket for placing the two legs of the coil, and the channels were patched by Stycast 1266 epoxy. Figure 1 shows the photograph of the setup. This immediately boosted the filling factor to 10^{-1}, and eliminated the background contribution significantly.

Although the technique was initially developed for detecting phase transitions, for instance superconducting or ferromagnetic transitions [10], I will show in this article that this microcoil method can be adopted for other microscopic measurements, such as the de Haas-van Alphen (dHvA) effect [1] and nuclear magnetic resonance (NMR) [6,7]

3. The de Haas-van Alphen effect

The existence of the Fermi surface is arguably the best signature of a crystalline, metallic state. The topography of the Fermi surface is predominantly mapped by two techniques: angle-resolved photoemission spectroscopy (ARPES) and quantum oscillations such as the dHvA effect. Unfortunately, it is impossible to carry out ARPES under pressure. Therefore, to probe the electronic structure of correlated electron systems under high pressure, the dHvA effect stands out as a powerful technique. In addition to gauging the size and the shape of Fermi surface sheets, this technique provides sheet-resolved quasiparticle effective masses. Impressive high pressure dHvA results have already been obtained on various heavy fermion systems using piston-cylinder pressure cells [11], providing a wealth of information for understanding the properties of these systems.

Very often, the phase of interest lies at the part of the pressure phase diagram that is too

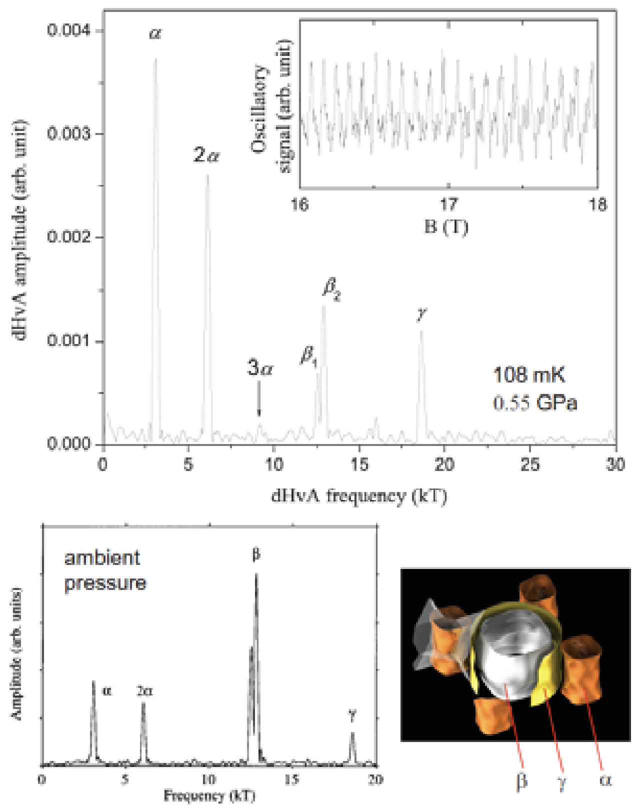

Figure 2. (Top panel) The dHvA signal of Sr_2RuO_4 and its FFT spectrum at 0.55 GPa, showing clearly all three branches of the Fermi surface. (Bottom panel) The ambient pressure spectrum, taken from [12], is shown for comparison. The Fermi surface of Sr_2RuO_4 is also displayed, showing the corresponding Fermi surface branches.

high for piston-cylinder pressure cells, which are limited to 3 – 4 GPa. Therefore, we are interested to perform dHvA measurements using an anvil cell, which can reach much higher pressures. As a first step, we investigated if it is feasible to perform the dHvA measurement using a 10-turn microcoil as a pickup coil.

Field modulation technique is a well-established method for the detection of dHvA signals [13]. A 140-turn copper coil was placed around the anvil, outside the sample chamber, to provide a modulation field of ~5 G. With this setup, clear dHvA signals have been observed for a few systems. Here we show the data obtained on a piece of ultra-clean Sr_2RuO_4 single crystal provided by Y. Maeno's group. The Fourier transform of our data reveals several peaks – three of them are fundamental frequencies that can be uniquely assigned to the α, β and γ sheets, in excellent agreement with previous ambient pressure dHvA works done under much favourable conditions (larger crystals, larger pickup coil with compensation scheme). In addition, the quasiparticle effective masses extracted by studying the temperature dependence of dHvA amplitudes are also consistent with earlier studies: $m^*/m_e = 3.4 \pm 0.1$, 7.0 ± 0.3 and 15.6 ± 1.2 for α, β and γ sheets, respectively [1]. Therefore, we believe that there is an opportunity to perform the dHvA effect at higher pressures using an anvil cell.

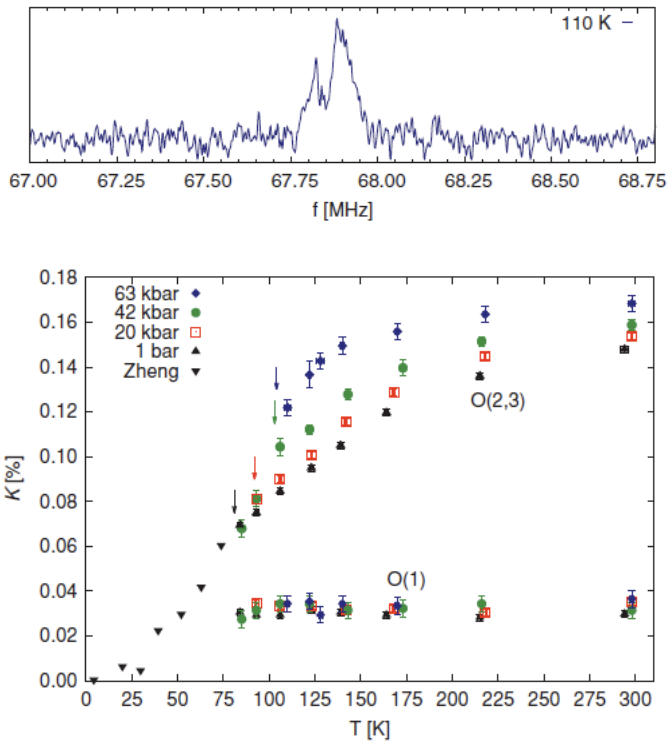

Figure 3. (Top panel) ^{17}O NMR spectrum of $YBa_2Cu_4O_8$ polycrystals at 6.3 GPa. (Bottom panel) Knight shift as a function of temperature at various pressures. The temperature independent shift comes from the apical O(1) nucleus, while O(2,3) denotes shifts from the planar oxygen sites. The arrows indicate the superconducting transition temperatures. The ambient pressure data from Ref. [14] are also shown for comparison.

4. Nuclear Magnetic Resonance

NMR is a very powerful tool to study the spin dynamics of correlated electron systems. To perform NMR experiments, a tunable *LC* resonator is constructed whose resonance frequency can be varied around the Larmor frequency of the probe nucleus under investigation. The microcoil naturally provides the inductance *L* necessary for the electrical resonator. On the other hand, the capacitive component is conveniently given by the feedthrough wire of the pressure cell, which acts like a cylindrical capacitor. Therefore, the necessary ingredients are in place to perform NMR experiments using anvil cells [6].

With this configuration, we have investigated various systems, including copper-based and iron-based superconductors, materials that might be useful for hydrogen storage, as well as simple elements. To illustrate the usefulness of the technique, I show the data collected on polycrystals of $YBa_2Cu_4O_8$ [7]. In copper oxide superconductors, it is well known that an anomalous pseudogap phase exists on the underdoped side of the phase diagram. Further doping the system closes the pseudogap. Applying pressure on underdoped cuprates raises the superconducting critical temperature, in similar manner to hole doping. Therefore, it is natural to ask if the pseudogap phase can be suppressed by pressure.

NMR Knight shift, a quantity which is proportional to the density of states, is a useful probe of the pseudogap phase. In the normal state, Knight shift has a non-Pauli-like temperature dependence, characteristic of a pseudogap behaviour. We performed ^{17}O NMR on $YBa_2Cu_4O_8$ polycrystals at various pressures. Following earlier works by Haase and coworkers [15,16], the Knight shift data shown in Figure 3 were analysed using the linear combination of two susceptibilities χ_1 and χ_2. It is found that the contribution from χ_1, which is temperature dependent, decreases steadily at high pressures, while the contribution from the temperature independent χ_2, increases by a factor of 9 at 6.3 GPa, the highest pressure reached for this study. Hence, pressure closes the pseudogap, and the large increase of the contribution from the Pauli-like susceptibility might explain why pressure enhances the superconducting critical temperature of $YBa_2Cu_4O_8$ so much.

In addition, we have studied elemental aluminium up to 10.1 GPa. We found a rapid suppression of the density of states at high pressure, which is consistent with a high pressure Lifshitz transition where some Fermi surface sheets disappear [17].

5. Mapping the phase diagram

As mentioned earlier, this technique was initially developed to detect ferromagnetic and superconducting transitions. Originally, the cell was configured in the *mutual inductance mode*, where a modulation coil similar to the configuration employed in Section 3 was in place. Using this configuration, we have recently constructed the high pressure phase diagram of $BaFe_2(As_{1-x}P_x)_2$, allowing the study of the interplay between chemical and physical pressures [3], and quasi-skutterudite compound $(Ca_{1-x}Sr_x)_3Ir_4Sn_{13}$, where we identified a superlattice quantum critical point beneath the superconducting dome [9].

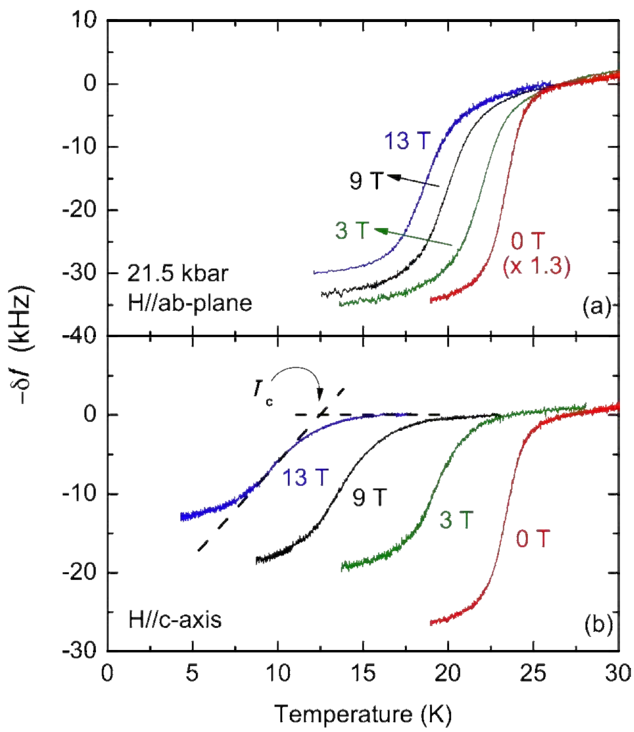

Figure 4. Temperature dependence of the resonant frequency of a high pressure resonator containing a single crystal of $BaFe_2(As_{0.65}P_{0.35})_2$ at 2.15 GPa with magnetic field applied along (a) the *ab* plane and (b) the *c* axis.

When we discussed the configuration for NMR experiments in the preceding section, we mentioned the need of building an *LC* resonator. In fact, we can also detect phase transition using the microcoil configured in the *resonator mode*, simply by tracking the resonant frequency of the resonator. The resonant frequency of a simple parallel *LC* circuit is given by $2\pi f = 1/\sqrt{LC}$. When a sample is inserted into a coil, its inductance is given by $L = L_0(1+\eta\chi)$, where L_0 is the inductance of the coil without any sample, η is the filling factor as defined in Section 2, and χ is the susceptibility of the sample. Therefore, when phase transition occurs, the resonant frequency alters correspondingly due to the change in χ. This technique is particularly sensitive to superconducting transitions, where the change in χ is of the order of unity, and the resonant frequency increases upon entering the superconducting state. We show in Figure 4 some example traces collected on $BaFe_2(As_{0.65}P_{0.35})_2$, a superconductor with a critical temperature of 31 K, where we studied the anisotropic superconducting properties as a function of pressure [4].

6. Future prospects and conclusions

The high pressure *LC* resonator described above operates in the radiofrequency range. Incidentally, the tunnel diode oscillator (TDO), a powerful method to probe the symmetry of the superconducting gap functions, also operates in the same frequency range. Naturally, one would like to ask if it is possible to incorporate a tunnel diode in series with our *LC* tank circuit, hence enabling the study of superconducting gap symmetry under pressure. This is indeed a subject for future investigations.

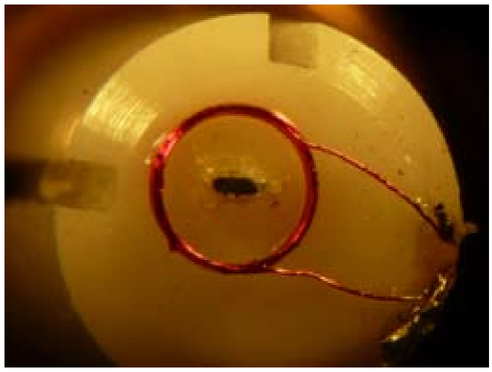

Figure 5. Photograph showing the interior of a uniaxial pressure cell. The crystal was polished to expose the crystallographic plane in such a way that pressure can be applied along the *ab* plane. The bottom piston is shown. The coil, which is slightly thinner than the crystal, has a diameter of 1.5 mm (Picture credit: H. Taniguchi)

So far, the article has only dealt with the situation where the pressure environment is hydrostatic, or at least quasi-hydrostatic. One could also apply force only along one particular direction, giving rise to *uniaxial* pressure effect. For low dimensional materials with anisotropic compressibility, uniaxial pressure can give drastically different results. To take an example, while hydrostastic pressure depresses the superconducting critical temperature of Sr_2RuO_4, uniaxial pressure enhances it [18].

Recently, in collaboration with H. Taniguchi and Y. Maeno of Kyoto University, a uniaxial pressure cell was designed to perform inductive measurements. A pickup coil was installed inside the pressure cell around the sample, thereby ensuring a good filling factor. It is hoped that in the near future, the high pressure experiments described in this article can also be carried out under uniaxial pressure.

In summary, I have discussed various aspects of inductive measurements currently being carried out under pressure using anvil cells. I have given examples from the de Haas-van Alphen effect, nuclear magnetic resonance as well as the mapping of the phase diagram using the microcoil technique. With suitable modifications, other physical properties, eg. penetration depth of superconductors, can also be measured under pressure, allowing detailed studies of exotic states that arise at high pressures.

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Curriculum Vitae

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